
Method and device for producing nitrogen fertilizer from
organic waste products

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SPECIFICATION

The invention relates to a method and a device for
producing nitrogen fertilizer from organic waste products
in the liquid phase (suspension, emulsion, solution) and
10 for hygienizing the wastes or reducing the emissions by
thermal treatment using mineral or organic additions.

Numerous methods for producing fertilizers from or-
ganic wastes such as fresh or fermented manure, animal
feces, muck, droppings or sewage sludge are known in the
15 art, which are directed to the production of the fertil-
izer as well as to the hygienization of the initial prod-
ucts and the reduction of foul-smelling substances and
harmful greenhouse gases, in particular ammonia.

For instance, DE 196 30 387 A1 and DE 101 20 372 A1
20 describe methods for producing a fertilizer from manure
with a considerably reduced release of ammonia, based on
the chemical reaction of manure with humus.

DE 43 04 342 C1 proposes a method for producing a
fertilizer on the basis of sewage sludge by mixing with
25 calcium sulfate. In DE 41 19 504 A1 is disclosed a method
for producing a combination fertilizer by mixing manure
and/or feces with gypsum. According to DE 44 44 726 C1, a
fertilizer is obtained by mixing manure with finely
ground fire clay and solidifying the resulting product.

In DE 196 44 613 C2, a pellet-type fertilizer from manure using CaO-containing residues from flue gas cleaning devices is proposed.

5 Further, methods such as in DE 40 33 509 A1 are known in the art, according to which liquid organic fertilizers and condenser water are mixed with biogenic substances such as straw and mineral substances such as gypsum so to obtain a fertilizer ready to spread, and are then subjected to a thermal re-treatment at 70 to 80 °C.

10 In DE 195 47 320 A1, a method and a device for producing an ammonia-free and sterile all-in-one fertilizer from biological wastes is described, wherein the fermented material is heated for at least 1 hour to the boiling temperature, and the released ammonia and other
15 volatile substances are removed together with the vapor. According to DE 42 43 918 A1, the fermented manure is boiled in a column, and the escaping ammonia-containing vapors are condensed in a mixing condenser with formation of ammonium salts.

20 All these methods are uneconomical and fulfill the objects of the method according to the invention to an incomplete extent only. The methods, which result in a fertilizer by mixing the waste products with additions, have to dry and finish the fertilizer in an expensive
25 way. Those methods, which drive ammonia in the boiling heat out, must solve the not simple object of condensation and water removal.

It is the object of the present invention to reduce the emissions of organic wastes, for instance manure,

with the simultaneous production of a high-quality nitrogen fertilizer and the conversion of the discharges, in particular in biogas systems, into an environmentally friendly, hygienically harmless sludge liquor by an economical and technically simple method.

According to the invention, this is achieved by a method using organic waste products in the liquid phase (suspension, emulsion, solution) by thermal treatment using mineral or organic additions, wherein the waste product is heated at underpressure to temperatures between 40 and 90 °C, the escaping gas containing carbon dioxide and ammonia is cooled down and introduced into an aqueous absorption agent or brought into contact therewith, the nitrogen fertilizer formed thereby is discharged and the excess gas not having been absorbed and containing carbon dioxide is pumped back into the discharge container, wherein the underpressure generated at the beginning of the process by a vacuum pump is autogenously maintained by the progress of the process.

Preferably, the excess gas not having been absorbed and containing carbon dioxide is pumped back into the cycle by either

- conducting it through the waste product to be treated, or
- immediately above the waste product to be treated, or
- through the gas cooling system above the waste product to be treated, or
- dividing it and conducting a partial flow through the waste product and another partial flow above the waste product.

Suitably, a temperature is adjusted in the gas cooling system, which is at least 3 K and at most 15 K below the temperature in the stripping container, whereas in the rear part another cooling-down process to 40 °C takes place.

It may be advantageous to add in addition carbon dioxide in a mixture with other gases, for instance from biogas plants, from outside to the excess gas conducted in the cycle.

It is advantageous to select a pressure of 10 to 70 kPa.

It has proven advantageous to first evacuate to 10 to 30 kPa, and to then increase the pressure to 40 to 80 kPa.

Further, it has turned out that the method is preferably suited for the treatment of animal excrements, such as manure and feces, in the liquid phase, without being limited thereto. The effect can also be transferred to further organic waste products, such as liquid muck and nitrogen-containing liquid wastes.

It has turned out as particularly advantageous, if fermented manure is used, to heat it up to 70 to 85 °C at a reduced pressure.

In this case, it may be advantageous to filter the fermented manure before its thermal vacuum treatment. The hygienized discharge manure remaining after the thermal treatment can then be sprayed on meadows and fields as a

virtually odorless sludge liquor stripped from nitrogen compounds. The solid substances separated by the filtration are well compostable.

5 A particularly suitable aqueous absorption agent is a gypsum suspension in water having a content of solid matter of 10 % by mass to 50 % by mass. It is favorable to stir this suspension and regularly remove the generated nitrogen fertilizer containing deposited lime and ammonium sulfate at the sump of the container. The fertilizer can immediately be used either as a thin mud or
10 also after drying as a crumbly mass or as a powder, or can be stored for later use.

In a preferred embodiment, the method according to the invention combines a degasification of the wastes in
15 a vacuum with a chemical reaction of the escaping gasses in a suspension of a finely ground mineral mass. Unexpectedly, the following surprising effect has been found: if the atmospheric pressure in a container filled with fresh or fermented manure of liquid muck is reduced with
20 simultaneous moderate heating to 40 to 90 °C, then first carbon dioxide, and at a slightly higher temperature ammonia will escape from the liquid, without being accompanied by appreciable amounts of water. When these gasses are conducted into a suspension of a finely ground mineral mass containing calcium compounds, they will react
25 under generation of lime and ammonium salts, but in a way that the underpressure is maintained, and the reaction is autogenously continued, if the temperature in the discharge container is maintained at the predetermined
30 level.

In this way, the ammonium nitrogen can nearly fully be removed, e.g. from a fermented manure. A virtually odorless sludge liquor is left, which does not gas any more, but still contains the mineral components of the manure, such as potassium and phosphate, in the form of its compounds. From the stirred suspension in the collector, the ready-to-use nitrogen fertilizer containing lime in addition to ammonium salts, can be taken out as a concentrated suspension. It can be used without any further re-treatment.

Since the gases fed into the collection container are not fully absorbed by the aqueous mineral suspension, they are returned into the stripping container and thus guided in a circulating course.

An acid solution, in particular a sulfate solution, is also suitable as an aqueous absorption product. Further, of course, a combination with a gypsum suspension in water is a suitable variant.

A clearly better result is obtained, if the gases fed into the collection container are returned to the stripping container and thus guided in a circulating course. This circulating course of the excess gas is an essential feature of the invention.

The device is composed of the following essential parts:

- a stripping container for heating at underpressure (1)

- a collection container for a reaction in a heterogeneous phase (2)
- a heat storage (thermosiphon) for heat exchange (3)
- a vacuum pump (4)
- 5 • a heating water pump (5)
- a circulation fan (6)
- a stirrer (7)
- and per se known pipelines, shutoff devices, and
- measurement and control devices.

10 A preferred embodiment of the device for producing nitrogen fertilizer consists in that

- the device comprises an additional gas cooling system with an upwards directed separating column and a downwards directed cooler, and
- 15 - additional pipelines and ball valves, in order that the circulating gas
- can be fed fully or partially into the stripping container above the waste product or
- through the cooling system into the collection container or
- 20 - partially into the stripping container into the waste product, wherein the residual flows in case of a division of the circulating gas are optionally fed into the two remaining designated entry positions.

25 The method and the device are characterized by low investment and operating costs and prevent risks for man and environment. The method has a minimum current consumption, since the underpressure in the system is maintained, once it has been generated by the vacuum pump.

The necessary process heat is substantially secured by the waste heat of a block heat and power plant of the biogas system connected upstreams. Further chemicals such as acids or lyes are not needed in the preferred embodiment of the invention. The system required for the method is easy to handle and can be operated as a batch process, but also continuously.

The invention is described in more detail by the following examples, and the device required therefor is explained by reference to three preferred variants (in Example 1) and two special cases (in Examples 2 and 3), without being limited thereto.

EXAMPLES

Example 1

Fig. 1 shows in an exemplary manner the scheme of
5 such a device for producing nitrogen fertilizer, as de-
scribed in Example 1 in more detail.

Therein are:

- 1 stripping container for heating at underpres-
sure
- 10 2 collection container for a reaction in the het-
erogeneous phase
- 3 heat storage (thermosiphon) for heat exchange
- 4 vacuum pump
- 5 heating water pump
- 15 6 circulation fan
- 7 stirrer
- 8 heat exchanger
- 9 inlet of discharge
- 10 outlet of heating water
- 20 11 return line for heating water
- 12 line for stripping gas
- 13 line for return gas
- 14 ball valve
- 15 ball valve
- 25 16 discharge residue
- 17 discharge nitrogen fertilizer
- 18 upwards directed part of the cooling system
(separation column)
- 19 downwards directed part of the cooling system
30 (cooler)

- 20-22 ball valves for return gas
- 23 shutoff valve

The system selected as Example 1 operates as a batch process. The heat exchanger (3), which is configured in an exemplary manner as a layer-type storage container (thermosiphon), serves for the intermediate storage of the heat of the treated discharge for heating-up the freshly poured-in liquid waste product, in the example 250 liters fermented manure (discharge) were used. The water in the heat storage is pre-heated by treated discharge and re-heated by the waste heat of a block heat and power plant over the heat exchanger (8), so that there is continuously a temperature of 90 °C at the storage head.

15 Start-up of the batch process.

The fresh discharge is poured into the stripping container (1) at position (9). After closing the container, heating-up is performed by means of heating water, which is taken up at position (10) directly at the head of the layer-type storage container, to a temperature of approx. 80 °C. The return flow of the heating water is fed again to the heat exchanger (3) through the line (11) by means of the heating water pump (5), and thus introduced again in the respective temperature zone.

25 After reaching the operating temperature, a pressure of 400 mbar is adjusted in the whole system by a controlled vacuum pump (4), and the pressure reduction is performed slowly and steadily. When this pressure has been obtained, the circulation fan (6) is set into opera-

tion, so that the stripping gas is sucked-off with a defined gas throughput from the stripping container (1) through the line (12), and return gas from the collection container (2) is fed to the stripping process through the line (13), and by means of the ball valves 20 to 22, three preferred variants of the circulation gas flow according to the invention can be selected:

Variant A: ball valve 21 is opened, whereas 20 and 22 remain closed. Then the circulation gas flows completely above the level of the waste product into the stripping container (1).

Variant B: ball valve 21 remains closed, and ball valves 20 and 22 are partially opened, so that part of the circulation gas flows through the waste product, whereas the remainder is returned between the column (3) and the cooler (4) into the process.

Variant C: ball valve 20 is opened, and ball valves 21 and 22 remain closed. Then the circulation gas completely flows back into the process at the indicated center of the cooling system.

Operating sequences of the batch process.

During the batch process, temperature and pressure in the stripping container (1) are held at approx. 80 °C and 400 to 500 mbar by switching the heating water pump (5) or the vacuum pump (4) on and off.

Under these conditions, in the course of approx. 2 hours, first CO₂ and then ammonia are driven out from the

discharge, reacted in the circulation process and washed-out in the following collector.

5 With the finely grained mineral mass being present there, for instance gypsum, the reaction products are reacted so to form lime and ammonium sulfate, and the undissolved mineral mass and lime particles are held in suspension by the stirrer (7). The stirring speed is to be selected such that no blocking of the inlets and outlets by the deposited lime occurs.

10 The mass of the driven-out ammonia is approx. 850 g per batch process. This mass reacts with approx. 3.4 kg gypsum, which has been suspended before in 10 l collection water.

15 As reaction products are generated approx. 2.5 kg lime and 3.3 kg ammonium sulfate in the liquid phase. Both substances can be used as a fertilizer without any further re-treatment.

Completion of the batch process.

20 After degassing the ammonia as far as possible from the discharge, the gas circulation driven by the circulation fan is set out of operation. The system is aerated.

25 Cooling-down of the treated discharge takes place, by closing the ball valve (14) and opening the ball valve (15) in the heating cycle, by conducting now cold storage water taken from the lower part of the heat storage through the heat exchanger of the stripping container. In this way, the heat of the treated discharge is trans-

ferred to the cold storage water. The heated-up storage water is in turn fed to the storage through the thermosiphon and introduced in the respective temperature zone into the heat storage.

5 After cooling-down of the treated discharge, the stripping container is emptied at the position (16) and is available for the next filling-up. After every batch process (before the new generation of vacuum for the next process), the sump is discharged at position (19) through
10 the valve (23) from the collection container, and a new gypsum suspension is introduced.

Example 2

15 The invention may also be carried out by the special case described in more detail in this Example 2, this case having the advantage of a slightly easier and faster execution, not being suitable, however, for all applications in an optimum manner.

20 Fig. 2 shows in an exemplary manner the scheme of such a special device for producing nitrogen fertilizer.

There are:

Full line:	gas circulation
Dotdashed line:	heating water circulation
Dashed line:	flows of substances

- 1 stripping container for heating at underpres-
- sure
- 2 collection container for the reaction in the
- heterogeneous phase
- 5 3 heat storage (thermosiphon) for heat exchange
- 4 vacuum pump
- 5 heating water pump
- 6 circulation fan
- 7 stirrer
- 10 8 heat exchanger
- 9 inlet of discharge
- 10 outlet of heating water
- 11 return line for heating water
- 12 line for stripping gas
- 15 13 line for return gas
- 14 ball valve
- 15 ball valve
- 16 discharge residue
- 17 discharge nitrogen fertilizer

20 The system of Example 2 shown as a special case
 again operates as a batch process. The heat storage (3),
 which is configured in an exemplary manner as a layer-
 type storage container (thermosiphon), serves for the in-
 termediate storage of the heat of the treated discharge
 25 for heating-up the freshly poured-in liquid waste prod-
 uct. In the example, 250 liters fermented manure (dis-
 charge) were used. The water in the heat storage is pre-
 heated by the treated discharge and re-heated by the
 waste heat of a block heat and power plant over the heat
 30 exchanger (8), so that there is continuously a tempera-
 ture of 90 °C at the storage head.

Start-up, operating sequences and completion of the batch process take place in an analogous manner to Example 1.

5 **Example 3**

This method is again carried out in a batch process according to the scheme shown in Example 1, however with the modifications described below.

10 The fresh discharge is poured into the stripping container (1) at position (9). After closing the container, heating-up is performed by means of heating water, which is taken up at position (10) immediately at the head of the heat storage container (3), to a temperature of 70 to 75 °C. The return flow of the heating water
15 is fed again to the heat storage (3) through the line (11) by means of the heating water pump (5), and thus introduced again in the respective temperature zone.

Before reaching the operating temperature, a pressure of > 400 mbar is adjusted in the whole system by a
20 controlled vacuum pump (4). After reaching the final pressure, the circulation fan (6) is set into operation, so that the stripping gas is sucked-off with a defined gas throughput from the stripping container (1) through the cooled line (12), and return gas from the collection
25 container (2) is fed to the stripping container through the line (13).

During the batch process, the temperature in the stripping container (1) is held constant at approx. 75 to 80 °C by switching the heating water pump (5) on and off, whereas the pressure slowly rises to 400 to 500 mbar.

5 Under these conditions, in the course of approx. 2 hours, first CO₂ and then ammonia are driven out from the discharge and washed-out in the following collector. In the collector, there is a suspension of 2.75 kg FDG gypsum in 8 liters water, which has been reacted with 2 li-
10 ters of a 20% sulfuric acid. The mass of the driven-out ammonia is approx. 850 g per batch process. As reaction products are generated approx. 2 kg lime and 3.3 kg ammonium sulfate in the liquid phase. Both substances can be used as a fertilizer without any further re-treatment.

15 The undissolved mineral mass and lime particles are held in suspension by the stirrer (7). The stirring speed is to be selected such that no blocking of the inlets and outlets by the deposited lime occurs.